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1	0	(decreas\$ or less\$) near20 aldehyde near20 polyester and hydride near transfer	USPAT; US-PGPUB; EPO; JPO; DERWENT; IBM_TDB	2004/08/05 09:04
2	4109	(decreas\$ or less\$) near20 aldehyde	USPAT; US-PGPUB; EPO; JPO; DERWENT; IBM_TDB	2004/08/05 09:05
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4	2175	(decreas\$ or less\$) near5 aldehyde	USPAT; US-PGPUB; EPO; JPO; DERWENT; IBM_TDB	2004/08/05 09:05
5	486	(decreas\$ or less\$) near5 aldehyde near5 (content or amount)	USPAT; US-PGPUB; EPO; JPO; DERWENT; IBM_TDB	2004/08/05 09:05
6	578	(decreas\$ or less\$) near5 aldehyde near5 (content or amount or concentration)	USPAT; US-PGPUB; EPO; JPO; DERWENT; IBM_TDB	2004/08/05 09:06
7	11	(decreas\$ or less\$) near5 aldehyde near5 (content or amount or concentration) near20 polyester	USPAT; US-PGPUB; EPO; JPO; DERWENT; IBM_TDB	2004/08/05 09:07
8	20	(decreas\$ or less\$ or small\$) near5 aldehyde near5 (content or amount or concentration) near20 polyester	USPAT; US-PGPUB; EPO; JPO; DERWENT; IBM_TDB	2004/08/05 09:44
9	0	(decreas\$ or less\$ or small\$) near5 aldehyde near5 (content or amount or concentration) near20 polyester and hydride	USPAT; US-PGPUB; EPO; JPO; DERWENT; IBM_TDB	2004/08/05 09:30

Day : Thursday

Date: 8/5/2004  
Time: 09:10:20 PALM INTRANET**Inventor Name Search Result**

Your Search was:

Last Name = RULE

First Name = MARK

Application#	Patent#	Status	Date Filed	Title	Inventor Name 51
<u>60564150</u>	Not Issued	020	04/21/2004	METAL PHOSPHONATES AND RELATED NANOCOMPOSITES	RULE, MARK
<u>60420984</u>	Not Issued	159	10/24/2002	POLYMERIC GEL FILTRATION MEDIA FOR REMOVAL OF VOLATILE, SEMI-VOLATILE, AND PARTICULATE ORGANIC AND INORGANIC MATERIALS FROM AIR AND STREAM	RULE, MARK
<u>10696858</u>	Not Issued	030	10/30/2003	PET COPOLYMER COMPOSITION WITH ENHANCED MECHANICAL PROPERTIES AND STRETCH RATIO, ARTICLES MADE THEREWITH, AND METHODS	RULE, MARK
<u>10693602</u>	Not Issued	020	10/24/2003	FILTERS AND METHODS OF MAKING AND USING THE SAME	RULE, MARK
<u>10664292</u>	Not Issued	093	09/17/2003	MULTILAYER POLYMERIC/INORGANIC OXIDE STRUCTURE WITH TOP COAT FOR ENHANCED GAS OR VAPOR BARRIER AND METHOD FOR MAKING SAME	RULE, MARK
<u>10664187</u>	Not Issued	093	09/17/2003	MULTILAYER POLYMERIC/ZERO VALENT MATERIAL STRUCTURE FOR ENHANCED GAS OR VAPOR BARRIER AND UV BARRIER AND METHOD FOR MAKING SAME	RULE, MARK
<u>10650150</u>	Not Issued	020	08/28/2003	POLYESTER COMPOSITION AND ARTICLES WITH REDUCED ACETALDEHYDE CONTENT AND METHOD USING VINYL	RULE, MARK

				ESTERIFICATION CATALYST	
<u>10621636</u>	Not Issued	071	07/17/2003	METHOD TO DECREASE THE ALDEHYDE CONTENT OF POLYESTERS	RULE, MARK
<u>10446094</u>	<u>6762275</u>	150	05/27/2003	METHOD TO DECREASE THE ACETALDEHYDE CONTENT OF MELT-PROCESSED POLYESTERS	RULE, MARK
<u>10393857</u>	Not Issued	020	03/21/2003	PACKAGED POTABLE LIQUID WITH UV ABSORBER FOR REDUCED OFF-TASTE FROM CLOSURE AND METHOD	RULE, MARK
<u>10233332</u>	Not Issued	041	08/30/2002	POLYESTER COMPOSITION AND ARTICLES WITH REDUCED ACETALDEHYDE CONTENT AND METHOD USING HYDROGENATION CATALYST	RULE, MARK
<u>10106503</u>	<u>6569479</u>	150	03/27/2002	PROCESS FOR REDUCTION OF ACETALDEHYDE AND OXYGEN IN BEVERAGES CONTAINED IN POLYESTER-BASED PACKAGING	RULE, MARK
<u>09909919</u>	<u>6599569</u>	150	07/20/2001	PLASTIC CONTAINERS WITH AN EXTERNAL GAS BARRIER COATING, METHOD AND SYSTEM FOR COATING CONTAINERS USING VAPOR DEPOSITION, METHOD FOR RECYCLING COATED CONTAINERS, AND METHOD FOR PACKAGING A BEVERAGE	RULE, MARK
<u>09846648</u>	<u>6632874</u>	150	05/01/2001	METHOD TO DECREASE ALDEHYDE CONTENT IN POLYOLEFIN PRODUCTS	RULE, MARK
<u>09817140</u>	Not Issued	164	03/27/2001	COMPUTER ASSISTED METHOD AND SYSTEM FOR ACCURATELY PREDICTING CO2 SHELF-LIFE OF POLYESTER CONTAINERS FOR CARBONATED BEVERAGES	RULE, MARK
<u>09735313</u>	<u>6548123</u>	150	12/12/2000	METHOD FOR COATING A PLASTIC CONTAINER WITH VACUM VAPOR DEPOSITION	RULE, MARK
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<u>09645639</u>	<u>6720052</u>	150	08/24/2000	MULTILAYER POLYMERIC/INORGANIC OXIDE STRUCTURE WITH TOP COAT FOR ENHANCED GAS OR VAPOR BARRIER AND METHOD FOR MAKING SAME	RULE, MARK
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<u>07822797</u>	<u>5143984</u>	150	01/21/1992	PROCESS TO PREPARE HIGH MOLECULE WEIGHT POLYESTER	RULE , MARK
<u>07819696</u>	<u>5180775</u>	150	01/13/1992	BLENDS OF COPOLY(ARYLENE SULFIDE) AND ETHYLENE- PROPYLENE RUBBER	RULE , MARK
<u>07536049</u>	<u>5021546</u>	250	06/11/1990	COPOLY(PHENYLENE SULFIDE)	RULE , MARK
<u>07522495</u>	<u>5026824</u>	150	05/14/1990	COPOLY(PHENYLENE SULFIDE)	RULE , MARK
<u>07270985</u>	Not Issued	168	11/14/1988	PROCESS FOR PREPARING ARYLTIO SUBSTITUTED AROMATIC COMPOUNDS	RULE , MARK
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<u>07232964</u>	<u>4855514</u>	150	08/17/1988	OXIDATIVE IODINATION OF PHENOL	RULE , MARK

<u>07228779</u>	<u>4866200</u>	150	08/04/1988	PROCESS FOR THE COPRODUCTION OF AROMATIC CARBOXYLATES AND ALKYL IODIDES	RULE , MARK
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<u>07219123</u>	<u>4952671</u>	250	07/15/1988	TERMINATED COPOLY (ARYLENE SULFIDE)	RULE , MARK
<u>07209120</u>	<u>4806676</u>	150	06/20/1988	CARBONYLATION PROCESS FOR PREPARATION OF AROMATIC CARBOXYLIC ESTERS AND AN ALKYL IODIDE	RULE , MARK
<u>07118572</u>	<u>4792600</u>	150	11/09/1987	PROCESS FOR THE PREPARATION OF COPOLY (ARYLENE SULFIDE) WITH AROMATIC NITRO COMPOUND CATALYST	RULE , MARK
<u>07115295</u>	Not Issued	166	11/02/1987	PROCESS FOR THE CO- PRODUCTION OF AROMATIC CARBOXYLATE AND ALKYL IODIDES	RULE , MARK
<u>07087332</u>	<u>4792634</u>	150	08/20/1987	PROCESS FOR THE PREPARATION OF ARYL SULFIDES	RULE , MARK
<u>07082300</u>	<u>4853480</u>	150	08/06/1987	OXYIODINATION INCORPORATING AN IODINE GENERATOR FOR THE CONVERSION OF METHYL IODIDE TO IODINE	RULE , MARK
<u>07078665</u>	<u>4814526</u>	150	07/28/1987	SELECTIVE ADSORPTION/SEPARATION OF DIODONAPHTHALENES	RULE , MARK
<u>07029897</u>	<u>4778940</u>	150	03/25/1987	PROCESS FOR PREPARING IODINATED SUBSTITUTED AROMATIC COMPOUNDS	RULE , MARK
<u>07002522</u>	<u>4847406</u>	150	01/12/1987	PROCESS FOR THE CO- PRODUCTION OF AROMATIC CARBOXYLATES AND ALKYL IODIDES	RULE , MARK
<u>07002521</u>	Not Issued	166	01/12/1987	PROCESS FOR THE CO- PRODUCTION OF AROMATIC CARBOXYLATES AND ALKYL	RULE , MARK

				IODIDES	
<u>06912806</u>	<u>4746758</u>	150	09/29/1986	PROCESSES FOR PREPARING IODINATED AROMATIC COMPOUNDS	RULE , MARK
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<u>06887488</u>	<u>4654436</u>	150	07/21/1986	CARBONYLATION PROCESS FOR THE PRODUCTION OF AROMATIC ACIDS OR ESTERS	RULE , MARK
<u>06801902</u>	Not Issued	161	11/26/1985	CARBONYLATION PROCESS FOR THE PRODUCTION OF AROMATIC ESTERS II	RULE , MARK
<u>06668130</u>	Not Issued	161	11/05/1984	PROCESS FOR MAKING MOLDED ARTICLES	RULE , MARK
<u>06517485</u>	<u>4446303</u>	150	07/26/1983	PROCESS FOR PREPARING HIGH MOLECULAR WEIGHT POLYESTERS	RULE , MARK
<u>06509112</u>	<u>4481314</u>	150	06/29/1983	INFRARED RADIATION ABSORBENT ANTHRAQUINONE DERIVATIVES IN POLYESTER COMPOSITIONS	RULE , MARK

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**PATENT ABSTRACTS OF JAPAN**(11)Publication number : **05-043782**(43)Date of publication of application : **23.02.1993**

(51)Int.Cl.

**C08L 67/02**  
**B29D 22/00**  
**B65D 1/09**  
**C08K 5/05**  
**// B29K 67:00**(21)Application number : **03-232322**(71)Applicant : **KURARAY CO LTD**(22)Date of filing : **19.08.1991**(72)Inventor : **OKAMOTO TOMOHIRO**  
**UNO MASANARI****(54) POLYESTER RESIN COMPOSITION FOR CONTAINER AND CONTAINER MADE THEREOF**

(57)Abstract:

PURPOSE: To provide a polyester resin composition for container, composed of polyethylene terephthalate and a higher alcohol and giving a container having low acetaldehyde content and suitable as a container for drink.

CONSTITUTION: The objective polyester resin composition for container is produced by compounding (A) 100 pts.wt. of a polyester resin containing ethylene terephthalate unit as main recurring unit and having an intrinsic viscosity of preferably 0.6-0.9dl/g with (B) 0.02-3 pts.wt. (preferably 0.1-2 pts.wt) of a higher alcohol, preferably a 10-35C saturated chain aliphatic alcohol (e.g. stearyl alcohol and cetyl alcohol) and, as necessary, a colorant, a filler, a lubricant, an ultraviolet absorber, etc. The addition of the component B is carried out e.g. by adding to a reaction system during the production of the component A or adding in the melt-molding of the component A. The obtained composition is molded to form a hollow vessel by extrusion blow-molding or injection blow-molding process, etc., or is extrusion molded to obtain a sheet, which is formed to a container such as tray.

**LEGAL STATUS**

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[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

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[Date of extinction of right]

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the container which consists of a polyester resin constituent for containers, and this resin constituent. It is related with the still more detailed polyester resin constituent for the containers with which this invention gives few polyester containers of an acetaldehyde content, and this container.

[0002]

[Description of the Prior Art] In recent years, the container obtained by carrying out melting shaping of the polyester resin including polyethylene terephthalate has appeared on the market in the commercial scene widely as bevel-use containers, such as carbonated drinks, such as cola, and mineral water, from the point of excelling in transparency, dynamics physical properties, health nature, and economical efficiency. However, the main polyester resin which is represented by polyethylene terephthalate and whose unit is ethylene terephthalate repeatedly is usually pyrolyzed at the time of the melting polymerization and melting shaping, and generates an acetaldehyde. If an acetaldehyde exists in the container quality of the material, in order to be eluted after contents restoration and to have a bad influence on the taste and smell of contents, it is the big evaluation criteria in the container which consists of the above-mentioned polyester resin that there are few amounts of acetaldehydes which exist in the polyester container quality of the material. Even for cola and mineral water tending [ very ] to be influenced by the acetaldehyde, and the elution of an acetaldehyde even especially spoiling the commodity value of these drinks remarkably, for a certain reason, the actual condition is that it is anxious for the polyester container with which the acetaldehyde content was reduced more.

[0003] There is the approach of using an object with few acetaldehyde contents as polyester resin used as the way method for reducing the amount of acetaldehydes in the container quality of the material which consists of main polyester resin whose unit is ethylene terephthalate repeatedly in order to carry out melting shaping of the container. In the purpose, it is known that it is effective to use the polyester resin obtained by carrying out solid state polymerization. That is, polymerization degree can be raised by \*\*\*\*\* which gives the polyester prepolymer obtained by the melting polymerization method to solid state polymerization under reduced pressure or circulation of inert gas, reducing the acetaldehyde content in this polyester prepolymer. However, depending on an application, it is hard to say the polyester resin obtained by this solid-state-polymerization method that the content of an acetaldehyde is level low not necessarily enough even if it is the container obtained by lying down on melting shaping, and a polyester ingredient for containers which gives a container with still still few contents of an acetaldehyde is desired.

[0004]

[Problem(s) to be Solved by the Invention] One of the purposes of this invention is to offer the polyester ingredient for containers which gives the polyester container with which the acetaldehyde content was reduced further, and other purposes of this invention are to offer fewer polyester containers of an acetaldehyde content.

[0005]

[Means for Solving the Problem] According to this invention, one of the above-mentioned purposes is repeatedly attained by offering the polyester resin constituent for containers which consists of higher alcohol of 0.02 - 3 weight section to the main polyester resin and this main polyester resin 100 weight section whose unit is ethylene terephthalate. Moreover, according to this invention, other above-mentioned purposes are attained by offering the container which consists of this polyester resin constituent.

[0006] the polyester resin in this invention -- as the principal component of a glycol component -- ethylene glycol -- desirable -- more than 70 mol % -- it contains. As for the ethylene glycol as one of the glycol components, as this ester plasticity derivative, ethylene oxide is illustrated here also including the ester plasticity derivative. It is desirable from giving the container the main copolymerized polyester resin repeatedly made into a unit excelled [ container ] polyethylene terephthalate resin or ethylene terephthalate in a mechanical property, transparency, etc. as this polyester resin. As a copolymerization component, from this viewpoint, preferably The phthalic acid of the amount not more than 10 mol % of all acid components, Isophthalic acid, hexahydrophthalic acid, naphthalene dicarboxylic acid, an adipic acid, dicarboxylic acid [ such as sebacic acid, ]; -- carboxylic acids, such as oxy acid like a p-oxy-benzoic acid, -- and -- desirable -- 1 of the amount not more than 10 mol % of all alcoholic components, and 2-propanediol -- 1,3-propanediol, 1,4-butanediol, 1,6-hexanediol, Diols, such as 1, 9-nonane diol, neopentyl glycol, a diethylene glycol, triethylene glycol, and cyclohexane dimethanol; alcohol, such as alcohol more than trivalent [ of trimethylol propane pentaerythritol, etc. ], is illustrated.



[0007] The polyester resin in this invention can be manufactured according to the usual approach. Namely, the glycol raw material which mainly consists of ethylene glycol; the polyester resin in this invention can be obtained by solidifying the prepolymer obtained [ low-grade polymer / the esterification reaction or / which was made to carry out an ester exchange reaction and was obtained ] by carrying out a melting polymerization in monomers, such as a dicarboxylic acid raw material which mainly consists of terephthalic-acid low-grade alkyl ester, such as a terephthalic acid or dimethyl terephthalate, considering as the chip of the configuration of arbitration, such as the shape of the shape of a dice, and a cylinder, and subsequently carrying out solid state polymerization of this chip. As for a melting polymerization, it is desirable under reduced pressure under existence of ester polymerization catalysts, such as diacid-ized germanium, to carry out on 250-290-degree C conditions. As for the limiting viscosity of the prepolymer generated by the melting polymerization, it is desirable that it is within the limits of 0.35 - 0.70 dl/g. In addition, in an esterification reaction, an ester exchange reaction, or a melting polymerization, diethylene-glycol byproduct inhibitors, such as organic amines, such as tetra-alkylammonium hydroxide, such as tetraethylammonium hydroxide, triethanolamine, and triethylamine, may be added in the system of reaction. The amount of acetaldehydes in a container may be further reduced by adding a diethylene-glycol byproduct inhibitor. After carrying out predrying of the prepolymer chip at the temperature of 190 degrees C or less, it is desirable to carry out solid state polymerization. As for solid state polymerization, it is desirable to carry out by making a prepolymer chip flow at the temperature of 190-240 degrees C under reduced pressure or circulation of inert gas, such as nitrogen gas. As for the limiting viscosity of the polyester resin obtained by solid state polymerization, in the purpose which obtains the good container of a mechanical property, it is desirable that it is within the limits of 0.6 - 0.9 dl/g.

[0008] In addition to the above-mentioned polyester resin, the polyester resin constituent of this invention contains higher alcohol. As this higher alcohol, the chain-like saturation fatty alcohol of the carbon numbers 10-35, such as stearyl alcohol, cetyl alcohol, and melissyl alcohol, is desirable. The content rate of the higher alcohol in the polyester resin constituent of this invention is 0.02 - 3 weight section to the polyester resin 100 weight section, and is 0.1 - 2 weight section preferably. The amount of higher alcohol cannot fully reduce the acetaldehyde content of the container obtained as it is under the 0.02 weight section to the polyester resin 100 weight section. The reduction effectiveness of the acetaldehyde content of the container which will be obtained on the other hand if 3 weight sections are exceeded falls, and the transparency of a container falls. The approach of arbitration, such as the approach of adding in the system of reaction during manufacture of (1) polyester resin as the combination approach into the polyester resin of higher alcohol and the approach of adding at the time of melting shaping of (2) polyester resin, is adopted. In addition, in the polyester resin constituent of this invention, different-species polymers, such as a well-known additive and/or polyethylene, such as a coloring agent, a bulking agent, lubricant, an ultraviolet ray absorbent, an anti-oxidant, and a surface active agent, a polycarbonate, and ethylene / vinyl alcohol copolymer, may contain.

[0009] The container of this invention can be manufactured by the melting fabricating method which may generally be used in usual polyester resin using the above-mentioned polyester resin constituent. For example, by blow-forming methods, such as the usual extrusion blowing-in method, the injection blowing-in method, and the approach (cold parison process) of carrying out blow forming, after reheating preforming, since it is filled up with drinks, such as a carbonated drink, juice, and mineral water, this polyester resin constituent can be used as a suitable hollow container. Moreover, after sheet-izing by extrusion molding, it can also consider as containers, such as a cup and a tray, by the vacuum or compressed-air thermoforming. In addition, it is desirable to fabricate in this melting shaping by low temperature and the short residence time generally, as much as possible [ more than the melting point of polyester resin and in order to control generation of the acetaldehyde in the time of melting shaping ], although the molding temperature of 320 degrees C or less is usually adopted.

[0010]

[Example] Hereafter, this invention is not limited by these examples although an example explains this invention concretely.

[0011] In addition, the measuring method adopted in the following examples of reference, examples, and examples of a comparison is as follows.

[0012] (1) Based on the result of having measured viscosity at 30 degrees C, it asked using the solution of the phenol / tetrachloroethane same weight mixed solvent of a limiting viscosity sample (a prepolymer or polyester resin).

[0013] (2) Preforming was formed with injection molding on conditions with a die temperature of 12 degrees C using the amount desiccation polyester resin chip of acetaldehydes in a hollow container for the barrel temperature of 290 degrees C, and cycle-time 36 seconds, then this preforming was heated, and the circular hollow container of 1.5l. of content volume was produced by carrying out blow forming on condition that the preforming temperature of 100 degrees C of just before blowing in, and blow pressure 20 kg/cm<sup>2</sup>. In addition, condominium PURASUTO LB01 was used as a blow-forming machine, using Nissei ASB50 as an injection molding machine. Thus, after sealing immediately after fabricating the obtained circular hollow container, leaving it at 21 degrees C and 65% of relative humidity for 1 hour and nitrogen gas's permuting the air in a container subsequently, it sealed and was left at 21 degrees C and 65% of relative humidity for 24 hours. The nitrogen gas in a container was collected, quantitative analysis was carried out by Shimadzu gas-chromatograph GC-9A, and it asked for the acetaldehyde concentration (mg/dl) of this nitrogen gas.

[0014] 100 ppm tetraethylammonium hydroxide was added to the slurry which consists of the example terephthalic-acid of reference 100 weight section, and the ethylene glycol 45 weight section, this was esterified at 250 degrees C under pressurization, and the mixture of screw-beta-hydroxyethyl terephthalate of 95% of rates of esterification and its low-grade polymer was prepared. Subsequently, the diacid-ized germanium 0.016 weight section as a catalyst was added to this mixture 124 weight section, the polycondensation reaction was performed at 280 degrees C under reduced pressure with an absolute pressure of 1

torr, and the prepolymer of limiting viscosity 0.60 dl/g was prepared, and it extruded in the shape of a strand, it cut from the nozzle, and was made the cylindrical chip. After drying the chip of the obtained prepolymer at 150 degrees C for 5 hours, solid state polymerization was carried out at 205 degrees C under the nitrogen air current for 15 hours. The limiting viscosity of the obtained polyester resin chip was 0.75 dl/g.

[0015] The polyester resin chip obtained in the example 1 and the example of 2 reference was supplied to the melting extruder, and after kneading with the stearyl alcohol of the amount which shows this polyester resin in Table 1 in a melting condition, injection molding was carried out to preforming. Subsequently, the circular hollow container with the good transparency of 1.5l. of content volume was obtained by carrying out blow forming of this preforming, respectively. The amount of acetaldehydes of this circular hollow container is shown in Table 1.

[0016] The polyester resin chip obtained in the example of the example reference of a comparison was supplied to the melting extruder, injection molding was performed, without blending stearyl alcohol, and the circular hollow container with the good transparency of 1.5l. of content volume was obtained by subsequently carrying out blow forming. The amount of acetaldehydes of this circular hollow container is shown in Table 1.

[0017]

[Table 1]

	ステアリルアルコールの 添加量 (重量部) (注)	得られた丸底中空容器の アセトアルデヒド量 ( $\mu\text{g}/\text{dl}$ )
実施例 1	0. 5 0	3. 2
実施例 2	1. 0 0	2. 7
比 較 例	0	4. 5

(注) ポリエステル樹脂 1 0 0 重量部基準

[0018]

[Effect of the Invention] According to this invention, the polyester resin constituent for containers which gives the container with which the acetaldehyde content was reduced is offered a passage clear from the above-mentioned example. Moreover, since there are few acetaldehyde contents, the container offered by this invention is suitable especially as a container for being filled up with the drink to which the taste or a smell tends to fall by acetaldehydes, such as cola and mineral water.

[Translation done.]